A PROCESS FOR THE PRODUCTION OF CELLULOSE FIBER PULP

Field of the Invention

The present invention is related to the field of production of cellulose fiber pulp. More specifically it is related to an "environmentally friendly" process that does not involve the use of harmful chemicals for the production of pulp from both agricultural waste and the traditional sources of cellulose fibers.

Background of the Invention

Cellulose fiber pulp for the production of paper and numerous other products is produced by a great variety of industrial processes. The pulp attained can be broadly classified onto three classes – mechanical, chemical, and biological – according to the principal steps in the process. This classification is useful but not always entirely accurate since most of the industrial processes require several steps of several types. For example, all of the chemical and biological processes begin with a mechanical step of breaking up the vegetative material into small pieces, e.g. chips, flakes, etc. in order to increase the surface area of the material and make the following chemical or biological step more efficient and economical.

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In the existing primarily mechanical processes, the means used to break up the material generally lead to breakage and damage of the cellulose fibers. This plus the presence of the lignin, that can not be separated from the cellulose by mechanical means, reduces the quality of the resultant pulp. Paper produced from such pulp is generally weak and discolors easily when exposed to light.

Traditional commercial chemical processes for the production of paper pulp from cellulose-containing material are associated with very high energy consumption and with severe environmental problems. The plant equipment is also often very complex and costly having to be built to withstand the corrosive effect of the chemicals used in the process, high pressures and temperatures that are used in some of the stages of the process, etc. The process emits foul smelling and often noxious gases and the water that is used and released at almost all stages of the processing contains large amounts of pollutants. In the worst cases, the gases and waste water are allowed to escape directly into the environment and, in the best cases, extremely expensive steps must be taken to collect and treat the exhaust gases and waste water before releasing them to the surroundings. It is also difficult and expensive to design an efficient cycle for reuse of the water within the plant. The main difficulty being that the drain water from one stage of the cycle often contains dissolved chemicals that interfere with the following steps and expensive processes are required to recover the chemicals from the water.

Much attention has been given to these problems over the years. Typically the result of this attention is to reduce the magnitude of the problem by introducing changes or improvements in the plant equipment and/or in the types of chemicals used to break down the plant material and release and process the cellulose fibers. Typical solutions of this type are presented in, for example, US 6,280,567. In this patent is also presented a review of the present state of the art in the field of cellulose pulp production. From the discussion in this patent it is evident that there is a need for simplifying the process of pulp production.

The most direct way to reduce the environmental problems associated with the production of paper pulp is to reduce as much as possible, or ideally to eliminate entirely, the use of chemicals in the process of reducing the vegetative matter to cellulose fibers. In such processes, the function of dissolving the lignin and freeing the cellulose fibers is carried out with enzymes instead of chemicals. An early example of such as process is disclosed in US 3,962,033.

Another environmentally related problem not discussed above relates to the source of vegetative material used for paper pulp production. Although

paper has always been and continues to be produced from a great variety of plant material, the essentially exclusive source for commercial production of pulp is trees. The continuing exploitation of the world's forests is related to climatic effects and has far reaching implications for the future of the planet. Many processes have been developed, especially in recent decades, for using alternate sources of plant material. These sources include plants grown specifically for the purpose as well as the parts of plants that remain after the harvest of the crop and that are normally considered to be agriculture waste.

An example of a system that combines the use of biological methods with the use of starting material that comes from plant material other than that from trees to produce cellulose pulp is revealed in US 6,379,495. This patent discloses a process for the production of enzymes for further use in a process of producing cellulose fibers from vegetable masses.

It is therefore a purpose of the present invention to provide a process for the production of pulp from cellulose-containing material that essentially eliminates the environmental problems of traditional commercial chemical processes.

It is another purpose of the present invention to provide a mechanical process for the production of pulp from cellulose-containing material that

provides pulp having cellulose fibers that are virtually undamaged relative to the fibers in pulp produced by traditional processes.

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It is still another purpose of the present invention to provide a process for the production of pulp from cellulose-containing material that reduces the energy and material consumption of traditional commercial chemical and biological processes by increasing the surface area of the vegetative material brought in contact with the chemical and biological materials.

It is yet another purpose of the present invention to provide a general process suitable for the production of pulp from all types of cellulose-containing material including agricultural waste and wood.

It is a further purpose of the present invention to provide a physical-biological-chemical process for the production of pulp from cellulose-containing material that is much more efficient than the methods of the prior art.

It is a still further purpose of the present invention to provide a physical-biological-chemical process for the production of pulp from cellulose-containing material that results in fibers of high quality.

Further purposes and advantages of this invention will appear as the description proceeds.

Summary of the Invention

The present invention is concerned with providing an "environmentally friendly" process for the production of cellulose fiber pulp that does not involve the use of harmful chemicals for the production of pulp from both agricultural waste and the traditional sources of cellulose fibers.

In a first aspect the invention provides a process for producing mechanical pulp from vegetative matter. The process comprises using high pressure fluid jets to break apart the vegetative matter into small particles and to further reduce the size of the particles by causing them to pass through one or a series of screens or gratings. Each screen or grating in the series comprising successively smaller openings than those of the previous one. The process produces pulp that is suitable for many applications.

In a second aspect the invention is extended by the addition of further mechanical and/or chemical and/or biological steps in order to attain delignified pulp.

The process can be carried out using wood as the starting material or, as is preferred, agricultural waste such as the stalks of cotton plants or sunflowers can be used. In the first step of the process the selected material is prepared for use by soaking it in water, if necessary, until the tissue of the vegetative material is thoroughly wet and for, some types of plants, removing the bark or outer layer/s of the stem. In the next step the raw material is broken apart into small pieces by using high pressure fluid (preferably water) jets and causing it to pass through a series of screens. The pieces are then placed in a reaction vessel together with water and a suitable inoculum where, with the aid of heat if necessary, biological breakdown of lignin takes place. Preferably, the cellulose fibers are then chemically treated to complete the delignification, bleached, sorted by diameter, cleaned, aligned, pressed into bales, and dried.

The present invention provides a process for the production of cellulose fiber pulp from vegetative matter comprising the following steps:

- a. preliminary preparation of the vegetative matter;
- b. initial physical breaking apart of the vegetative matter by using high pressure fluid jets to break apart the matter and causing it to pass through a series of screens or gratings, wherein each screen or grating in the series contains successively smaller openings; and

c. biological delignification of the vegetative matter, accomplished by placing the vegetative matter together with water and an inoculum, into a reaction vessel.

Preferably, the process further comprises:

- a. chemical delignification, bleaching, and cleaning;
- b. initial sorting of cellulose fibers by diameter;
- c. final sorting and alignment of fibers;
- d. pressing the sorted and aligned fibers into bales; and
- e. drying the bales.

In the process of the invention, the preferred source of vegetative matter is agricultural waste. Such agricultural waste can comprise any part of agricultural plants from the group comprising but not limited to: cotton, corn, banana, sunflowers, watermelon rinds, wheat, and other cereal crops or grasses.

The preliminary preparation of the vegetative matter can include removing the bark or outer layer/s of the stem and soaking the matter in water. In some circumstances inoculum is added to the vegetative matter and water at the preliminary stage. The preferred fluid for use in the initial physical breaking apart of the vegetative matter is water. The pressure of the water used for initial physical breaking apart of the vegetative matter is between 200 and 1500 atmospheres and the openings in the screens are essentially square ranging between 1 and 20 mesh. In a preferred embodiment of the invention, the series of screens comprises three screens having essentially square openings of 1 mesh, 5 mesh, and 15 mesh respectively. It should be noted that additional, higher mesh screens can be added to the series thereby reducing the size of the particles to essentially single fibers.

According to the invention the inoculum comprises biological material that is unique for each type of vegetative matter. The process of biological delignification can be aided by heating the contents of the reaction vessel up to a temperature of 65°C. The process of biological delignification can be further aided by stirring the contents of the reaction vessel by means of a mechanical stirrer and/or streams of gas or water. The biological delignification can be carried out continuously by periodically removing the essentially delignified fibers from the reaction vessel and replacing the removed portion with more vegetative matter, water, and inoculum.

Preferably chemical delignification and bleaching is carried out by using a form of active oxygen, for example, stabilized hydrogen peroxide, preferably at a concentration of 20% to 50%, or oxygen gas.

The initial sorting of cellulose fibers by diameter is accomplished by causing them to pass through a series of screens, wherein each screen in the series contains successively smaller openings. In a preferred embodiment of the invention, the series of screens consists of four screens having essentially square openings of 25 mesh, 50 mesh, 75 mesh, and 100 mesh respectively. In other embodiments, the openings are slits (orientated either parallel to or perpendicular to the long axis of the screen).

The final sorting and orientation of the fibers are carried out by causing the fibers to pass through gratings. In a preferred embodiment of the invention, the spacing between adjacent "bars" of the gratings (i.e. the slit width) is in the range of $20\mu M$ to $300\mu M$.

The sorted and aligned fibers are pressed into bales using a pressure of 20Atm to 400Atm and air dried at a temperature of 30°C to 70°C.

All the above and other characteristics and advantages of the invention will be further understood through the following illustrative and non-limitative description of preferred embodiments thereof, with reference to the appended drawings. The process of the invention is carried out by using specially designed apparatus to carry out a series of well defined steps for reducing the vegetative raw material into dried blocks of fine cellulose pulp.

Brief Description of the Drawings

- Fig. 1A is a flowchart showing the steps in the process of the invention;
- Fig. 1B schematically shows the apparatus used to carry out the principal steps of the process of the invention;
- Fig. 2 is a schematic drawing showing the equipment used, according to a preferred embodiment of the invention, for the original breaking apart of the organic material;
- Fig. 3 is a schematic drawing showing the reaction vessel, according to a preferred embodiment of the invention;
- Fig. 4A is a schematic drawing showing a perspective view of the sizing equipment, according to a preferred embodiment of the invention;
- Fig. 4B is a schematic drawing showing a front view of the sizing equipment, according to a preferred embodiment of the invention;
- Fig. 5A is a schematic drawing showing a perspective view of the equipment for aligning the fibers, according to a preferred embodiment of the invention; and
- Fig. 5B is a schematic drawing showing a front view of the equipment for aligning the fibers, according to a preferred embodiment of the invention.

<u>Detailed Description of Preferred Embodiments</u>

The process of the invention is carried out by using specially designed apparatus to carry out a series of well defined steps for reducing the vegetative raw material into dried blocks of fine cellulose pulp. Fig. 1A is a flowchart showing the steps in the process of the invention, up to the stage of obtaining the delignified cellulose pulp. As is discussed herein, steps 1(optional) and 2 define a complete mechanical process that results in high quality, although not delignified, cellulose pulp suitable for the production of certain types of paper as well as other products. The mechanical pulp produced by the process of the invention can also be used with no further processing as "starting" material for a chemical or biological delignification procedure to produce high quality delignified cellulose pulp. If the pulp produced is to be sorted by fiber size of stored, then any or all of the steps 4, 5, and 6, described hereinbelow can be added to the process.

Fig. 1B schematically shows the apparatus of the production line used to carry out steps 2 to 5 of the process of the invention.

As will be illustrated in the examples discussed hereinbelow, the process of the invention is general, and with suitable changes in the values of the process parameters, can be adapted for use with virtually any type of cellulose containing material from the wood of trees to the stalks of wheat or other cereal crops or grasses. The preferred raw material for use in the

invention is agricultural waste, such as the stalks or stems of crops such as cotton, corn, banana, and sunflower. These crops are grown on a large scale and the stalks are normally left in the field to be burned, plowed-under, or otherwise disposed of after the harvest. As a very rough indication of the potential of this source of cellulose pulp we can consider the contribution of cotton. In Israel approximately 15,000 hectares of cotton are planted each year. After harvest there remains an estimated 200,000 tons (dry weight) of stem material per hectare. Using an estimated yield considerably lower than that achieved in the example described below, this amount of stem material will yield, using the process of the invention, at least 30,000 to 40,000 tons of cellulose pulp, i.e. approximately 10% of the amount annually used by the paper industry in Israel.

In the preferred embodiment of the invention it is preferable, but not obligatory, that the vegetative material used in the process be wet. Thus step 1 of the process is concerned with initial cleaning of the raw material to remove foreign objects, stones, dirt, etc. and insuring that the material possesses the required level of wetness. Material that is brought to the processing line directly from the field may contain sufficient moisture to allow it to be used directly. Stored material, or material that has dried out in the field, is placed into tanks filled with water for a period that typically ranges from several hours to several months depending on the material and its condition. For some types of plants, the bark or outer layer/s of the stem

may not be suitable for producing fibers of the proper quality for a particular application; therefore these layers must be removed at the beginning of the process. The crops are harvested during short periods once (or a few times) a year and the vegetative matter must be stored for latter processing. It can be stored dry or, preferably, can be stored in water in closed containers until needed. If desired, and especially if the waiting time will be long, inoculum can be introduced into the containers and biological delignification of the vegetative material can begin.

After suitable preparation, the raw material is transported to the equipment shown in section 2 of Fig. 1B and shown enlarged in Fig. 2. In Fig. 2 are shown three stations 20a, 20b, and 20c. Each of these consists of a box-like construction which supports a strong wire screen 21a, 21b, and 21c near its upper surface and a conveyor belt 28 near the bottom to convey the material that passes through the screen to the next station. The three screens have increasingly smaller, essentially square, openings ranging from 1 to 20 mesh. In a preferred embodiment of the invention, 21a is 1 mesh, 21b is 5 mesh, and 21c is 15 mesh. In other embodiments of the invention, the openings are slits having slit widths corresponding to the dimensions of the square openings, i.e. a grating comprising parallel wires (bars) with a spacing of 1 to 20 bars per inch.

Wet raw material from step 1 is placed on the screen 21a. A fluid at high pressure is then caused to impact on the material in order to initially break it up into smaller pieces, increasing its surface area. In the preferred embodiment of the invention, water from a reservoir 22 is pumped through pipes 23 using a pump (not shown in the figures) capable of delivering water at high pressure and having a high supply rate. The water system is equipped with valves 24, pressure gauges 25, pressure reducers 26, and high pressure nozzles 27. Water sprayed through the nozzles 27 at high pressure impacts upon the material on the screen and breaks it apart into pieces of diameter small enough to pass through the screen. These pieces fall onto the conveyor belt and are transported to station 20b, where they are split into smaller diameter pieces that are transported to station 20c. At 20c they are split again, finally exiting from this stage and entering the next stage of the process. The pressure used ranges between 200-1500 atmospheres, can be individually adjusted for each station, and depends on the type of vegetative matter used and its condition after the first step of pre-conditioning. The water passes through the bottom of each of the stations passing through, for example a 100 mesh sieve and is collected in tray 29 from which it exits through valve 24b and is returned through valve 24a to reservoir 22 where it is reused. This description of the path of the water cycle is merely illustrative and it may be that instead of returning directly to reservoir 22, it is first reused at other steps of the process. At all stages of this description, the references to the fact that the water returns to valve 24a, are not necessarily to be taken literally, but rather to serve to indicate that all the water is filtered and recycled through an appropriately designed closed water system with none of the water used in the process exiting the processing plant.

It should also be noted that for each of the screens of the production line used to carry out the process of the invention, appropriate methods are provided for clearing away (manually or automatically) the material that is too large and will not pass through a particular screen and remains on the top. These methods, as well as the automatic control system for controlling all the parameters of each stage of the process and also the pumps, motors, conveyor belts, etc. of the system, are well known to the person of experience and therefore will not be described herein for brevity. The conveyor belts can also be made of screen material with appropriate openings so that some of the separation or filtering steps of the process are carried out as the material is being transported between stations in the process. It should be noted that in this description conveyor belts are mentioned as a method of transporting the material from place-to-place, however alternate methods can also be used. For example, the process is extremely well suited for transport of the vegetative material suspended in water via chutes or by pumping through pipes.

The process described hereinabove i.e. the use of high pressure fluid and screens with openings of decreasing size results in a gradual breaking apart of the plant tissue without damaging the fibers composing the tissue. In the prior art, the processes used for braking up the plant material amounts to essentially grinding or shredding of the tissue, causing extensive random damage to the fibers. In the process of the invention, on the other hand, the plant tissue is separated into small assemblages of cellulose fibers essentially without causing them severe damage. Further the cellulose fibers produced by the process of the invention are not weakened by the chemicals used (in the later steps of the process) in the prior art. This result is achieved because the increased efficiency of the chemical reaction that results from the increased surface area of the particles allows for the use of lesser quantities of chemicals than required by prior art methods.

The process as described hereinabove leads to the production of mechanical pulp that is suitable for production of paper and other products. The resulting pulp can pass through the steps of sorting, pressing into bales, and drying that are described hereinbelow with reference to Figs. 4A to 5B.

The pulp resulting from the process of the invention described hereinabove can be delignified using chemical or biological means. A particular advantage of the method of the invention for initially breaking apart the vegetative material described hereinabove is that if the material is now introduced into a reaction vessel, where a chemical or biological delignification process is to take place, it has a surface area that is greatly increased over that of starting material prepared by prior art methods. The large surface area, combined with the fact that the pieces of the material are approximately of the same relatively small size, results in a greatly increased efficiency of biological and chemical breakdown of the material.

A mainly biological process will now be described for delignification of the mechanical pulp. The process described is nonlimitative and is chosen to illustrate the invention. As stated hereinabove, and will be readily understood by skilled persons, the mechanical pulp produced by the method of the invention is suitable for starting material in any delignification process. The material that passes through screen 21c is now transported to the reaction vessel shown at 3 in Fig. 1b and enlarged in Fig. 3 where most of the lignin will be biologically broken down leaving mainly the cellulose fibers. Reaction vessel 30 is a double-walled tank with a lid 38 which allows it to be sealed if necessary. Near the top of the tank are a number of openings and valves 34 through which materials such as the partially broken apart organic matter, water, inoculum, etc. can be introduced into the reaction vessel. On the sides near the bottom of the vessel are several smaller valves 35. These are used to introduce jets of water or gases to aid in stirring the mixture. At the bottom of the tank are large valves 36 used for draining contents of the vessel. As shown in the figure, arrangements are made for recycling the water (arrow to valve 24a), passing the cellulose fibers to the next step of the process (arrow to piece of equipment 40), etc. Inside the tank is stirring rod 31. Rod 31 can be moved back and forth along track 33 or rotated about its longitudinal axis by means of motors 32. The reaction vessel also comprises heating means 37, symbolically shown as a heating coil at the lower part of the tank. The heating means are capable of maintaining the temperature of the contents of the reaction vessel at a maximum of approximately 70°C if necessary. The heating can be accomplished by any suitable arrangement such as circulating heated water in the hollow between the walls of the vessel or by using an electrical heating element either in direct contact with the contents of the reaction vessel or enclosed between the walls. In a preferred embodiment of the invention, the water used to heat the reaction vessel is heated by solar energy.

The broken apart material, which has been split by the use of high pressure into pieces of a diameter small enough to pass through screen 21c, is now introduced into the reaction vessel 30 along with water heated to a temperature of up to 65°C (depending on the type of material) in the approximate ratio of 1 part vegetative matter to 7 parts water. The inoculum is now added to the contents of the reaction vessel. The inoculum comprises microorganisms that break apart the lignin bands surrounding the celluloid fibers and produced by standard techniques.

As mentioned hereinabove, the breaking up of the raw material in the previous step of the process of the invention (resulting in a greatly increased surface area) whether taking place at room temperature (25°C) or, optionally, with heating of the mixture in the reaction vessel to 35°C to 65°C (depending on the heat tolerance of the microorganisms) greatly accelerates the rate of the biological activity.

After initial mixing, the contents of the reaction vessel are left for a period of one day to several months until most of the lignin is removed. During this period, the mixture is intermittently gently stirred, either by activation of the stirrer or by introducing streams of gas or water through valves 35. The stirring action helps the biological breakdown of the organic material by providing flow of the liquid and also by breaking up the larger pieces of plant material.

After the biological breakdown, the water is drained from the reaction vessel, filtered through an appropriate screen (for example 130 mesh), and returned to the reservoir to be reused. In the preferred embodiment of the invention, some form of active oxygen (i.e. O₂, H₂O₂, OH⁻, O₃, etc.) is now added to the vegetative material. The purpose of the active oxygen is to complete (chemically) the delignification of the vegetative matter and to bleach the resulting cellulose fibers. The active oxygen is left in the reaction

vessel for a period of 20 to 200 hours to complete the final separation of the fibers and to bleach them.

As described hereinabove, the bleaching step is carried out in the reaction vessel used for the biological dissolution of the lignin. If required by considerations of efficient organization of the production schedule, the organic matter can be transferred to another container and the bleaching step can be carried out therein. As the biological delignification process proceeds, the delignified fibers tend to rise to the top the heavier pieces of matter sink to the bottom. In the preferred embodiment of the invention, this situation is taken advantage of by supplying a mechanism to periodically remove the essentially delignified fibers from the reaction vessel while leaving the partially delignified pieces behind for a longer time. As some of the material is removed, more vegetative material, water, and inoculum are added thus allowing for continuous operation of the biological process. In Fig. 3, the removal mechanism is schematically represented by device 39. This device can be, for example, a rake that is dragged across the reaction vessel pulling the contents to a port (not shown) from which they exit the reaction vessel and are transported to a container for the bleaching step of the process.

It will be clear to the skilled person that other methods of bleaching that do not involve the use of corrosive or dangerous chemicals can be employed, e.g.

causing a stream of oxygen to bubble through an aqueous solution of the cellulose fibers.

Following the bleaching process, the fibers are thoroughly rinsed with water which is then drained off, filtered and returned to the storage tank. The fibers are transferred to the equipment shown at section 4 in Fig. 1B. This equipment is used to sort the fibers by the diameter of the fibers. The sorting equipment is shown schematically in perspective view in Fig. 4A and in front view in Fig. 4B.

Referring to Fig. 4B, the sorting equipment 40 comprises four hoppers 41a, 41b, 41c, and 41d supported one above the other by frame 42. At the bottom of each leg of the frame is a spring 43 which symbolically represents a vibration system capable of vibrating sorting equipment 40 in the directions shown by orthogonal arrows 44, i.e. the vibration is in both the horizontal and vertical directions.

The bottom of each hopper is composed of a screen comprising essentially square openings (as described above for a previous step in the process, the openings can be slits having appropriate slit widths). The opening in the screens decreases from the uppermost to the lowest hopper. In a preferred embodiment of the invention the bottoms of hoppers 41a, 41b, 41c and 41d are screens of 25, 50, 75, and 100 mesh respectively. The fibers are

fibers are retained on the screen of 41a and the smaller ones drop through to 42b where some are retained and the remainder drop through to 42c, etc. The sorting by size is aided by the vibration of the equipment. A shower of water from an arrangement similar to that described with respect to Fig. 2, but operated at lower pressure is used to aid the screening process. As in all parts of the system, all water is collected by a suitable arrangement shown symbolically in Fig. 4B by tray 29 with the water exiting in the direction of valve 24a, the entrance to the water reservoir.

Frame 42 is constructed such that the longitudinal axes of the hoppers are at an angle with respect to the horizontal. The vibration of the equipment will cause the fibers that are to big to pass through a particular screen to move along the screen until they fall off the lower end and onto a conveyor belt 28. Each of the four conveyor belts moves its respective portion to the next piece of processing equipment.

It is to be noted that the size of the openings in the screens and the number of hoppers, i.e. the number of fractions into which the fibers are divided, can easily be either increased or decreased, depending on the size of fiber that is required for the manufacture of specific products from pulp produced by the method of the invention.

The equipment designed for carrying out the next stage of the process (step 5 in Fig. 1A) is schematically shown in perspective in Fig. 5A and in front view in Fig. 5B. Referring to Fig. 5B, each of the fractions resulting from the sorting is conveyed to one of four bins 51a, 51b, 51c, and 51d. Near the top of each bin is a grating 52a, 52b, 52c, and 52d respectively and near the bottom a conveyor belt 28 that leads the fibers from each size group to the final stages of the process.

The spacing between adjacent "bars" of the gratings (i.e. the slit width) is in the range of 20µM to 300µM and is selected to be appropriate for the diameter of the fibers in each of the respective groups. When the fibers fall through the grating they are all aligned with their longitudinal axis parallel to the direction of the slits in the grating. In addition the grating "filters out" any oversized fibers that have somehow passed through the earlier sorting. Analogously to sorting equipment 40, the bins 51a, 51b, 51c, and 51d are mounted on frame 54 equipped with vibration means 54. An overhead watering system is also provided to aid in passing the fibers through the grating.

After final sorting and alignment, the fibers of each of the four groups are conveyed separately to a conventional press and drying equipment. At this stage they are pressed using a pressure of 20Atm to 400Atm into bales of pulp. The bales are air-dried at a temperature of 30°C to 70°C.

Examples

The following examples are provided merely to illustrate the invention and are not intended to limit the scope of the invention in any manner.

Example 1: soft material

Raw material: 10 kg dry wheat straw

Step 1: soak in water two weeks

Step 2: screens -1, 5, and 15 mesh

pressure - 150-250Atm

Step 3A: time – two to four months

temperature - 37°C

stirring - mechanical stirrer

Step 3B: bleach material – stabilized hydrogen peroxide (35%)

time - 24 hours

Step 4: screens -25, 50, 75, and 100 mesh

Steps 5 and 6: yield (dry weight) -4Kg

Example 2: medium hard material

Raw material: stems of cotton plants (10kg dry weight equivalent)

Step 1: stems wet, no soaking necessary, remove outer layer of stem

Step 2: screens -1, 5, and 15 mesh

pressure – 250-500Atm

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Step 3A: time -six months

temperature -room temperature

stirring - stream of water

Step 3B: bleach material – stabilized hydrogen peroxide (35%)

time -one week

Step 4: screens - 25, 50, 75, and 100 mesh

Steps 5 and 6: yield (dry weight) - 5Kg

Example 3: hard material

Raw material: pine wood (10kg dry weight equivalent)

Step 1: wet - no soaking necessary; remove outer layer of bark

Step 2: screens - 1, 5, and 15 mesh

pressure - 500 - 1500Atm

Step 3A: time -eight months

temperature - ambient temperature

stirring - stream of water

Step 3B: bleach material - stabilized hydrogen peroxide (50%)

time -one week

Step 4: screens - 25, 50, 75, and 100 mesh

Steps 5 and 6: yield (dry weight) -7 Kg

The pulp obtained by the method of the invention is similar to cellulose pulp obtained by the methods of the prior art. As described above, the result of

the process is bales of dried cellulose pulp. This pulp can be used as the starting material for the manufacture, using conventional-well known methods, of all the products produced from cellulose fibers such as paper, fiber board, etc. The process of the invention, because it can be applied to all types of potential starting material and also because of the sorting of the fibers into fractions, wherein each fraction contains fibers with a relatively narrow range of diameters, allows for the production of pulp suitable for all applications.

Although embodiments of the invention have been described by way of illustration, it will be understood that the invention may be carried out with many variations, modifications, and adaptations, without departing from its spirit or exceeding the scope of the claims.

Claims

1. A process for producing mechanical pulp from vegetative matter, said process comprising using high pressure fluid jets to break apart said vegetative matter into small particles and to further reduce the size of said particles by causing them to pass through one or a series of screens or gratings, each comprising successively smaller openings than those of the previous screen or grating in said series.

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- 2. A process according to claim 2, further comprising the step of preliminary preparation of the vegetative matter prior to breaking apart said vegetative matter.
- 3. A process according to claim 1 further comprising the steps:
 - a. initial sorting of particles by diameter;
 - b. final sorting and alignment of the particles;
 - c. pressing the sorted and aligned particles into bales; and
 - d. drying the bales.
- 4. A process according to claim 1, wherein the vegetative matter is agricultural waste.

- 5. A process according to claim 4, wherein the agricultural waste comprises parts of agricultural plants from the group comprising, but not limited to:
 - a. cotton;
 - b. corn;
 - c. banana;
 - d. sunflower;
 - e. watermelon rinds;
 - f. wheat; and
 - g. other cereal crops or grasses.
- 6. A process according to claim 2, wherein the preliminary preparation of the vegetative matter includes removing the bark or outer layer/s of the stem.
- 7. A process according to claim 2, wherein the preliminary preparation of the vegetative matter includes soaking said matter in water.
- 8. A process according to claim 1, wherein the fluid is water.
- 9. A process according to claim 8, wherein the pressure of the water is between 200 and 1500 atmospheres.

- 10.A process according to claim 1, wherein the openings in the screens are essentially square ranging between 1 and 20 mesh.
- 11. A process according to claim 1, wherein the gratings comprise parallel wires (bars) with a spacing of 1 to 20 bars per inch.
- 12. A process according to claim 1, wherein the series of screens comprises three screens having essentially square openings of 1 mesh, 5 mesh, and 15 mesh respectively.
- 13. A process according to claim 3, wherein initial sorting of particles by diameter is accomplished by causing them to pass through a series of screens or gratings, wherein each screen or grating in said series contains successively smaller openings.
- 14. A process according to claim 3, wherein the final sorting and orientation of the particles are carried out by causing said particles to pass through gratings.
- 15. A process according to claim 3, wherein the sorted and aligned particles are pressed into bales using a pressure of 20Atm to 400Atm.

- 16. A process according to claim 15, wherein the bales are air dried at a temperature of 30°C to 70°C.
- 17. A process according to claim 1, comprising further mechanical and/or chemical and/or biological steps for the delignification of the small particles of vegetative matter.
- 18. A process according to claim 17, comprising biological delignification of the vegetative matter, accomplished by placing said vegetative matter into a container containing water and an inoculum of lignin degrading microorganisms.
- 19. A process for the production of cellulose fiber pulp from vegetative matter according to claim 17 comprising additionally some, or all of the following steps:
 - a. preliminary preparation of said vegetative matter;
 - b. chemical delignification, bleaching, and cleaning;
 - initial sorting of cellulose fibers by diameter;
 - d. final sorting and alignment of fibers;
 - e. pressing the sorted and aligned fibers into bales; and
 - f. drying the bales.

- 20. A process according to claim 18, wherein the preliminary preparation of the vegetative matter includes soaking said matter in water containing inoculum.
- 21. A process according to claim 18, wherein the process of biological delignification takes place in a reaction vessel.
- 22. A process according to claim 21, wherein the process of biological delignification is aided by heating the contents of the reaction vessel.
- 23. A process according to claim 22, wherein the heating is from room temperature up to a temperature of 65°C.
- 24. A process according to claim 21 wherein the process of biological delignification is aided by stirring the contents of the reaction vessel.
- 25. A process according to claim 24, wherein the stirring is carried out by means of a mechanical stirrer and/or streams of gas or water.
- 26. A process according to claim 21, wherein the biological delignification is carried out continuously by periodically removing the essentially delignified fibers from the reaction vessel and replacing said removed portion with more vegetative matter, water, and inoculum.

- 27.A process according to claim 19, wherein stabilized hydrogen peroxide is used for chemical delignification and bleaching.
- 28. A process according to claim 19, wherein initial sorting of cellulose fibers by diameter is accomplished by causing them to pass through a series of screens or gratings, wherein each screen or grating in said series contains successively smaller openings.
- 29. A process according to claim 28, wherein the series of screens consists of four screens having essentially square openings of 25 mesh, 50 mesh, 75 mesh, and 100 mesh respectively.
- 30. A process according to claim 28, wherein the series of gratings consists of four gratings comprising essentially parallel wires (bars) with a spacing of 25 to 100 bars per inch.
- 31. A process according to claim 19, wherein the final sorting and orientation of the fibers are carried out by causing said fibers to pass through gratings.

- 32. A process according to claim 31, wherein the spacing between adjacent "bars" of the gratings (i.e. the slit width) is in the range of $20\mu M$ to $300\mu M$.
- 33. A process according to claim 19, wherein the sorted and aligned fibers are pressed into bales using a pressure of 20Atm to 400Atm.
- 34. A process according to claim 19, wherein the bales are air dried at a temperature of 30°C to 70°C.
- 35. Cellulose pulp produced according to the process of claim 1 or claim 17.
- 36. A product manufactured from cellulose pulp produced according to the process of claim 1 or claim 17.

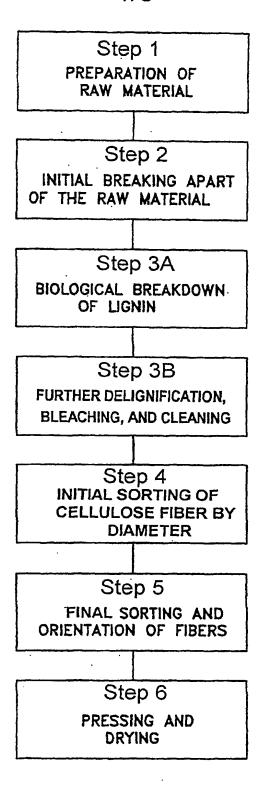
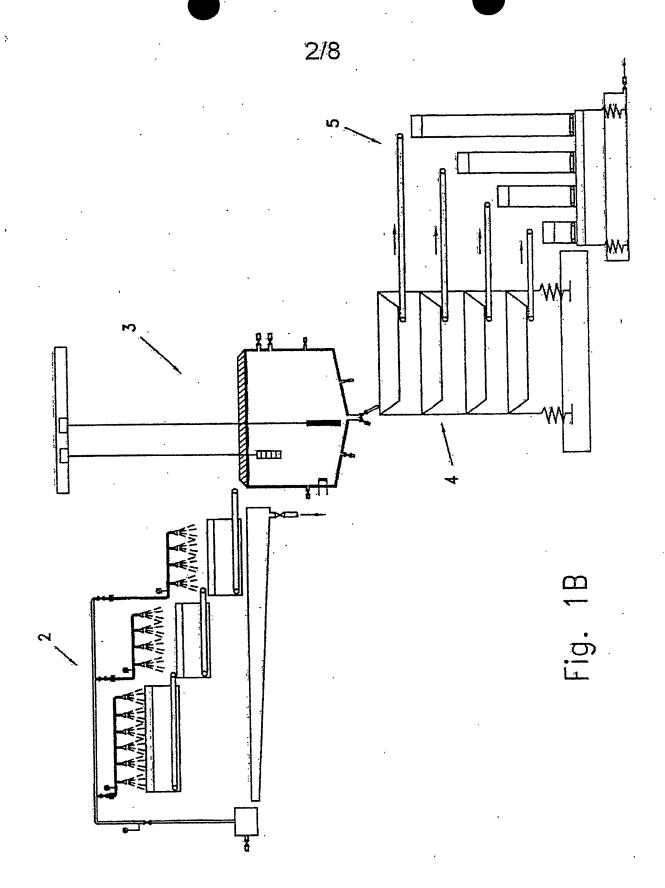
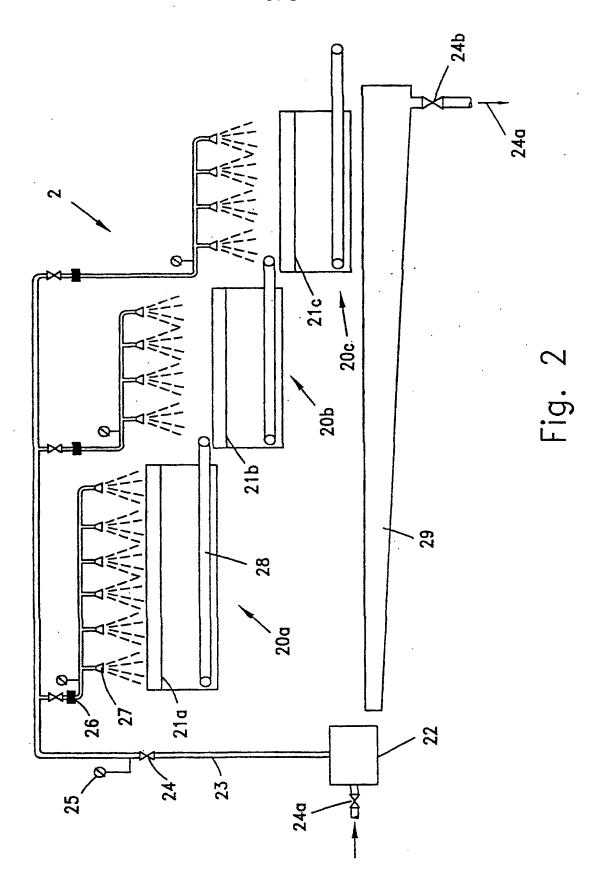
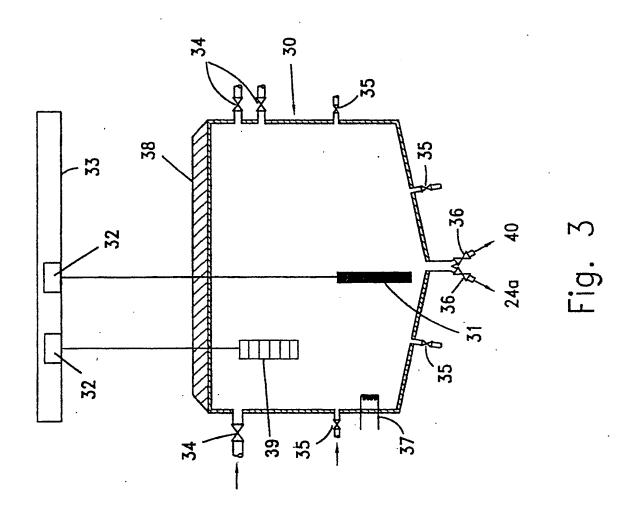


Fig. 1A







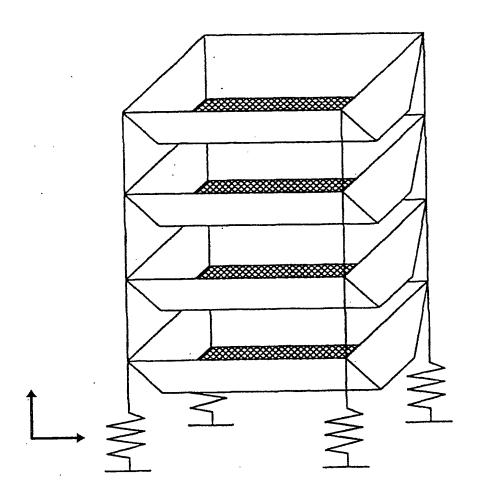


Fig. 4A

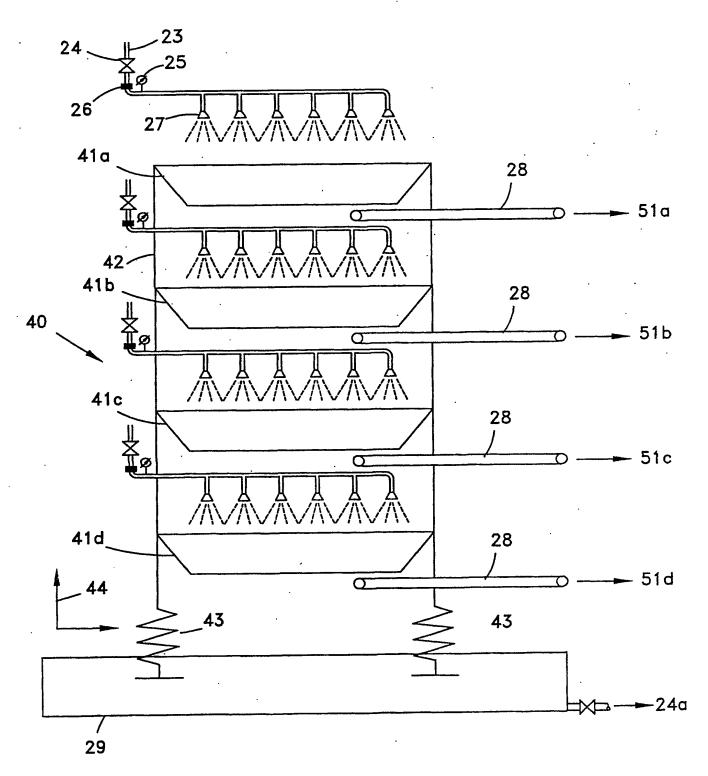
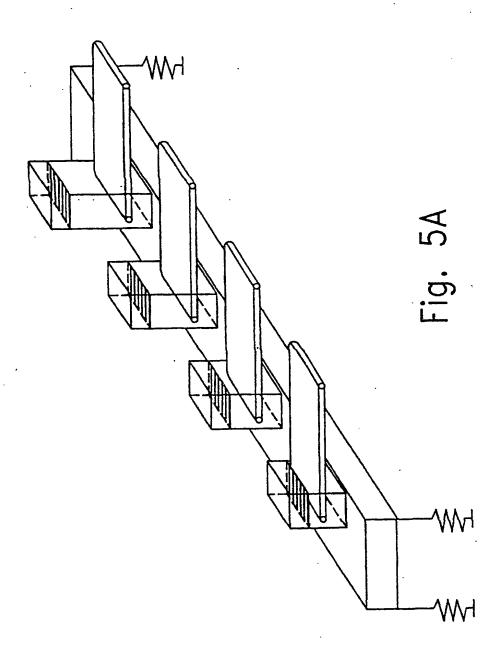


Fig. 4B



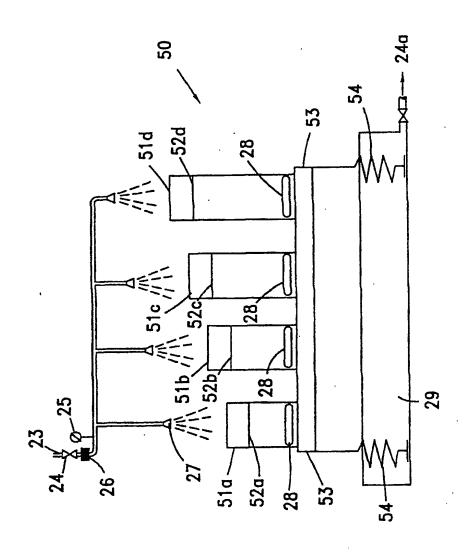


Fig. 5B

INTERNATIONAL SEARCH REPORT

PCT/I 3/00537

A. CLASSIFICATION OF SUBJECT MATTER IPC 7 D21C5/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols) IPC 7 D21C D21B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the International search (name of data base and, where practical, search terms used)

EPO-Internal, PAJ, WPI Data

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DE 197 54 766 A (LANGE GUENTER ; GRAUPNER GERALD (DE)) 20 January 2000 (2000-01-20)	1,2,4,8, 17,18, 21-23, 35,36
	page 3, line 6-11; claims 1,2	
X	US 3 326 607 A (NILS BOOK) 20 June 1967 (1967-06-20) claims 1-8; figures 1,16	1,2,4, 35,36
X	US 4 831 127 A (WEIBEL MICHAEL K) 16 May 1989 (1989-05-16) claims 1-10; example 9	1,2,4, 17,35,36
X	US 3 620 911 A (EKLUND HENRIK J ET AL) 16 November 1971 (1971-11-16) claims 1-14; example 1	1,2,4, 17,35,36
	-/	

X Further documents are listed in the continuation of box C.	Patent family members are listed in annex.
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Date of the actual completion of the international search	Date of mailing of the international search report
14 October 2003	27/10/2003
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INTERNATIONAL SEARCH REPORT

Internation No PCT/ 3/00537

C.(Continu	ation) DOCUMENTS CONSIDERED TO BE RELEVANT	37 00337		
Category °		Relevant to claim No.		
A	US 4 923 981 A (MYERS CHESTER D ET AL) 8 May 1990 (1990-05-08) the whole document	1-36		
A	US 5 035 362 A (MAZURKIEWICZ MARIAN) 30 July 1991 (1991-07-30) the whole document	1-36		
Α	US 5 008 254 A (WEIBEL MICHAEL K) 16 April 1991 (1991-04-16) the whole document	1-36		
A	US 6 379 495 B1 (CAPPELLLETTO PIER LUIGI ET AL) 30 April 2002 (2002-04-30) cited in the application the whole document	1–36		

Information on patent family members PCT/ 3/00537 Publication Patent family Patent document Publication member(s) date cited in search report date DF 19754766 A1 20-01-2000 DE 19754766 Α 20-01-2000 24-02-1969 308854 B US 3326607 Α 20-06-1967 SE BE 658534 A CH 422041 A 15-10-1966 DE 1287916 B DK 113222 B 03-03-1969 FI 41335 B 30-06-1969 1050746 A GB 6500678 A 22-07-1965 NL 31096 T 15-12-1987 Α 16-05-1989 ΑT US 4831127 30-04-1987 561116 B2 ΑU ΑU 1850583 A 08-03-1984 BR 8304803 A 10-04-1984 27-10-1987 CA 1228558 A1 DE 07-01-1988 3374703 D1 401583 A ,B, DK 04-03-1984 EP 0102829 A1 14-03-1984 ES 8506839 A1 16-11-1985 01-05-1987 ES 8703555 A1 30-11-1986 IL 69623 A 23-03-1995 JP 1914212 C 25-05-1994 JP 6039482 B JP 09-05-1984 59080402 A 9103908 B1 15-06-1991 KR 28-09-1990 MX 161464 A NONE US 3620911 Α 16-11-1971 15-02-1994 100675 T US 4923981 Α 08-05-1990 ΑT AU 614751 B2 12-09-1991 AU 1765088 A 15-12-1988 CA 1334920 C 28-03-1995 DE 10-03-1994 3887389 D1 DE 3887389 T2 01-09-1994 EP 21-12-1988 0295865 A2 01-07-1994 ES 2051851 T3 JP 1086845 A 31-03-1989 AT 31096 T 15-12-1987 30-04-1987 ΑU 561116 B2 ΑU 1850583 A 08-03-1984 BR 8304803 A 10-04-1984 CA 1228558 A1 27-10-1987 DE 07-01-1988 3374703 D1 401583 A ,B, DK 04-03-1984 ΕP 0102829 A1 14-03-1984 ES 8506839 A1 16-11-1985 8703555 A1 01-05-1987 ES 30-11-1986 IL 69623 A 23-03-1995 JP 1914212 C JP 25-05-1994 6039482 B JP 59080402 A 09-05-1984 KR 9103908 B1 15-06-1991 MX 161464 A 28-09-1990 US 5008254 A 16-04-1991 US 4629575 A 16-12-1986

INTERNATIONAL SEARCH REPORT

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pplication No

INTERNATIONAL SEARCH REPORT

Information on patent family members

Internation plication No
PCT/I 3/00537

				FC1/1 3/0053/	
Patent document cited in search report		Publication date		Patent family member(s)	Publication date
US 5035362	Α	30-07-1991	US	4723715 A	09-02-1988
US 5008254		16-04-1991	AT	146190 T	15-12-1996
			ΑU	630755 B2	05-11-1992
			AU	6564190 A	09-05-1991
			CA	2029022 A1	02-05-1991
			DΕ	69029365 D1	23-01-1997
			DE	69029365 T2	10-04-1997
			DK	426434 T3	30-12-1996
			EP	0426434 A1	08-05-1991
			ES	2098257 T3	01-05-1997
			GR	3022784 T3	30-06-1997
			JР	3197502 A	28-08-1991
			ΑT	31096 T	15-12-1987
			ΑU	561116 B2	30-04-1987
			ΑU	1850583 A	08-03-1984
			BR	8304803 A	10-04-1984
			CA	1228558 A1	27-10-1987
			DE	3374703 D1	07-01-1988
			DK	401583 A ,B,	04-03-1984
			EΡ	0102829 A1 ´	14-03-1984
			ES	8506839 A1	16-11-1985
			ES	8703555 A1	01-05-1987
			ΙL	69623 A	30-11-1986
			JP	1914212 C	23-03-1995
			JP	6039482 B	25-05-1994
			JP	59080402 A	09-05-1984
			KR	9103908 B1	15-06-1991
			MX	161464 A	28-09-1990
			US	4923981 A	08-05-1990
			US	4629575 A	16-12-1986
US 6379495	B1	30-04-2002	IT	MI960160 A1	31-07-1997
			ΑT	206778 T	15-10-2001
			DE	69707261 D1	15-11-2001
			DE	69707261 T2	02-05-2002
			DK	877839 T3	19-11-2001
			EP	0877839 A1	18-11-1998
			CA	2244464 A1	07-08-1997
			WO	9728306 A1	07-08-1997
			ES	2162239 T3	16-12-2001
			PT	877839 T	28-02-2002
			US	2002100570 A1	01-08-2002

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